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MICROSTRUCTURAL AND TRIBOLOGICAL ANALYSIS OF T4-TREATED Al6061-ZrB_2 COMPOSITES FABRICATED BY STIR CASTING

Aluminum Metal Matrix Composites (AMMCs) are well known for their superior mechanical properties and wear resistance, which makes them suitable for use in automobiles and the aerospace industry. Wear behavior, tensile strength and micro-Vickers hardness of Al 6061 reinforced with ZrB_2, produced by the stir casting process, were studied in this research. To further improve its characteristics, the composite was subjected to T4 heat treatment, consisting of solutionizing, quenching, and natural aging. Experimental findings indicated noticeable improvements in tensile strength, hardness as well as wear resistance with higher ZrB_2 content. The increased hardness is due to strong interfacial bonding between Al 6061 matrix and ZrB_2 particles and precipitation hardening effect due to T4 treatment. Additionally, the homogeneous dispersion of ZrB_2 particles efficiently resisted material loss during wear. The results indicate that Al 6061/ZrB_2 composites, especially T4-treated ones, highly suitable for applications requiring superior surface hardness and durability.

Keywords: Aluminum Metal Matrix Composites; T4 heat treatment; stir casting; wear

1. Introduction

The requirement for high-performance engineering materials with enhanced mechanical and tribological properties has been consistently growing, especially in aerospace, automotive, and defense applications. Such applications demand materials with high strength-to-weight ratio, good wear resistance, and thermal stability under harsh operating conditions. Traditional metals and alloys, though common, tend to be inadequate for these applications as they soften, wear, or lose structural integrity at high temperatures [1,2].

Metal matrix composites (MMCs) have also been a potential substitute as they could provide the toughness and ductility of metals combined with the hardness and thermal stability of ceramic reinforcements. Aluminum-based MMCs are particularly desirable owing to aluminum's low weight, good corrosion resistance, and ease of fabrication. These kinds of composites are presently used in key components like engine blocks, pistons, brake rotors, and structural aerospace components where weight savings and performance integrity are issues of primary importance [3].

Al6061 alloy is among the most used matrices for MMCs, providing good corrosion resistance, formability, and thermal conductivity [4]. Nevertheless, its mechanical property and wear resistance decrease under conditions of high-temperature services, restraining it from direct application in harsh environments. In order to overcome such limitations, different ceramic reinforcements have been explored in previous research. For instance, aluminum oxide (Al_2O_3) has been found to enhance hardness, silicon carbide (SiC) increases strength and wear resistance that enhances nucleation and microstructural refinement [5,6]. Even though these reinforcements enhance some properties, their deficiencies at harsh operating conditions – like oxidation susceptibility or poor interfacial bonding – make it necessary to explore other reinforcements.

Zirconium diboride (ZrB_2) is a newly identified very promising reinforcement material for aluminum MMCs. As a member of the ultra-high-temperature ceramics, ZrB_2 shows high hardness, good thermal and electrical conductivity, low density, and excellent oxidation and wear resistance. Most importantly, ZrB_2 reveals good wettability and strong interfacial bonding with alu-

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minum, which are essential for load transfer and microstructural stability [7]. In comparison to the traditional reinforcements such as SiC and Al_2O_3, ZrB_2 presents better high-temperature behavior and wear and fracture resistance, making it a choice candidate for advanced MMC evolution. Although the above encouraging qualities are presented, research on Al6061–ZrB_2 composites is still relatively small, and there is an evident gap in research [8].

The current investigation is focused on synthesizing Al6061–ZrB_2 composites and testing their mechanical and wear properties under dry sliding. The study is aimed at the influence of reinforcement content and applied load on wear rate and tribological behavior. Microstructural observation of fractured and wear-tested samples is utilized to evaluate matrix-particle interfacial bonding and its influence on performance determination. The results indicate the potential of Al6061–ZrB_2 composites as advanced materials for aeronautics and automobile industries, in which lightweight, high-strength, and wear-resistant materials are not optional.

2. Materials and methods

The Al6061 alloy was selected as the matrix material for the fabrication of the composite, with its chemical composition outlined in TABLE 1. Nano-sized ZrB_2 powder was utilized as the reinforcement due to its high hardness, thermal stability, and ability to enhance the mechanical properties of aluminum-based composites . The fabrication process began with melting the Al6061 alloy in an electric furnace at 750°C . To improve wettability and reduce surface tension, magnesium chips were added as a catalyst, followed by Hexachloroethane (C_2Cl_6) as a degassing agent to eliminate dissolved gases and impurities from the molten alloy . Before the addition of the reinforcement, degasser tablets were introduced into the melt to remove slag and impurities from the crucible .The ZrB_2 nano powder was preheated to 400°C for 15 minutes in a muffle furnace under an argon atmosphere to avoid oxidation and moisture absorption. After preheating, ZrB_2 nanoparticles in varying weight percentages of 0 wt.%, 0.5 wt.% and 1 wt.% were gradually added to the molten Al6061 alloy during the formation of a vortex in the melt, which was achieved through mechanical stirring at 750°C. Stirring facilitated the uniform dispersion of the nanoparticles, improving the bonding with the aluminum matrix. The composite in molten form was poured into a metal die (200 mm in length and 20 mm in diameter) under an argon atmosphere to minimize oxidation during solidification. The fabricated specimens were designated as TC0(Al6061), TC1(Al6061-0.5 wt.% ZrB_2) and TC2 (Al6061-1 wt.% ZrB_2) based on the ZrB_2 content ranging from 0% to 1% reinforcement.

TABLE 1

Chemical composition of Al6061

Element	Mg	Fe	Cu	Mn	Si	Al
Composition (%)	0.69	0.23	0.31	0.33	0.52	Balance

2.1. T4 heat treatment

The T4 heat treatment process for Al6061-ZrB_2 composites includes solution heat treatment, quenching, and natural aging to optimize mechanical properties and maintain good formability[9]. During solution treatment at 530-550°C, alloying elements like Mg and Si dissolve into the aluminum matrix, while ZrB_2 particles aid in grain refinement . Rapid quenching in water traps these elements in a supersaturated state, preventing premature precipitation and preserving ZrB_2's structural integrity due to its high melting point (~3245°C) . Natural aging at room temperature promotes the gradual formation of Mg_2Si precipitates, enhancing hardness and strength [10]. The addition of ZrB_2 improves load-bearing capacity and wear resistance due to its high hardness (~22 GPa) and thermal conductivity, making the composite ideal for demanding applications. This synergistic effect leverages Al6061's lightweight nature and ZrB_2's exceptional properties to enhance overall mechanical performance.

2.2. Instrumental analysis

The composites were mechanically polished on emery paper with grits ranging between 500 and 2000. The samples were etched using Keller's reagent solution after polishing for revealing their microstructure. Microstructural analysis was carried out using a scanning electron microscope (SEM, Hitachi S-3700N, Japan). The tensile test was conducted in accordance with ASTM E8 standards [11]. Hardness analysis was performed using an automatic turret micro-Vickers hardness tester (HV-1000ZDT, India). A 300 g load at a dwell of 15 seconds was employed. For repeatability and precision, the reading for each sample was recorded as the average of five readings.

A pin-on-disc (POD) machine was employed to examine the wear behavior of Al6061 and its ZrB_2-reinforced composite MMCs. The experiments were conducted between imposed loads of 20 N and 40 N with a 20 N step change. The cylindrical test samples of Al6061 and Al6061-ZrB_2 MMCs of dimensions 10 mm diameter and 40 mm height were used according to ASTM-G99 [12]. These test samples were studied under dry slide conditions against EN31 hardened steel counter face of hardness 62 HRC. For experiments, EN31 steel disc rotated at a constant speed of 520 rpm with constant velocity of sliding as 1.57 m/s.

During the tests, the height loss due to wear was measured at 30-second intervals using a linear variable differential transformer (LVDT) with an accuracy of 2.0 μm. This allows for precise monitoring of the wear in microns. To further investigate the wear mechanisms and the type of fracture in both the alloy and the composite specimens, optical microscopy (OM) analysis was performed on the worn-out surfaces. The OM images provided insights into the abrasive, adhesive, and delamination wear patterns, correlating the microstructural features with the observed wear characteristics, as previously reported in related studies .The EDX was performed to evaluate the elements present on the wear surfaces and to evaluate the formation of any intermetallic.

3. Results and discussions

3.1. Microstructure analysis

Microstructure refinement due to homogeneous particle distribution improving hardness. In the case of Al6061-ZrB_2 composites under T4 treatment, the ZrB_2 particles act as effective nucleation sites, refining the grain size during solidification shown in Fig. 1. This refinement reduces *d*, the average grain diameter, increasing the grain boundary area. Grain boundaries act as obstacles to dislocation movement, increasing the resistance to deformation.

When the average grain diameter (*d*) decreases, the total grain boundary area within the material increases because more, smaller grains mean more boundaries per unit volume. These grain boundaries act like barriers or obstacles that block the movement of dislocation defects in the crystal structure responsible for plastic deformation [13].

Dislocation motion is the primary way metals deform under stress. When grain boundaries obstruct dislocations, it becomes harder for the metal to deform, which means the material exhibits greater strength and hardness. In other words, the material resists deformation better because dislocations must navigate around or pile up at these boundaries, requiring higher applied stress to continue deforming. This is the fundamental basis of grain boundary strengthening or the Hall-Petch effect, where finer grains lead to improved mechanical properties by limiting dislocation mobility.

Since microhardness is directly related to yield strength, the Hall-Petch relationship predicts an increase in microhardness with decreasing grain size [14]. The observed ~15% increase in microhardness at 1 wt.% ZrB_2 reinforcement aligns with this theory, as the refined grains enhance the grain boundary strengthening effect. Thus, the ZrB_2-induced grain refinement leads to a finer microstructure, which increases the hardness by impeding dislocation motion, consistent with the Hall-Petch effect.

3.2. Tensile strength and micro hardness

The fabricated composites were subjected to tensile loading on universal testing machine (UTM) and the following values were obtained. the highest UTS was obtained in the sample TC2 consisting of 1 wt.% ZrB_2 having the value of 181.72MPa. This is attributed to the microstructural refinement caused by ZrB_2 in the composite. The next highest UTS(156.24 MPa) was observed in TC1 with 0.5 wt.% ZrB_2. The uniform dispersion of the reinforcement in Al6061 matrix leads to effective load transfer which barricades the dislocation motion resulting in enhanced tensile strength. The least UTS was displayed by TC0 which is pure Al6061 with the value of 139.89 MPa. This indicates that adding ZrB_2 reinforcement positively influenced the tensile properties of the composites. Also, there has been increase in the tensile strength of the composites with increasing concentration of ZrB_2. However, the effect on the % elongation has been contrasting with the increasing ZrB_2 concentration within the composites. The % elongation exhibited by TC0 (7.65%), TC1 (6.43%), TC2 (5.21%) is proof of the same. This is because, the presence of rigid ceramic particles like ZrB_2 introduces localized stress concentrations and limits the material's ability to deform plastically [15]. Therefore, as the wt.% of ZrB_2 increases, the aluminum matrix becomes more constrained, reducing its capacity to absorb strain, which slightly compromises ductility.

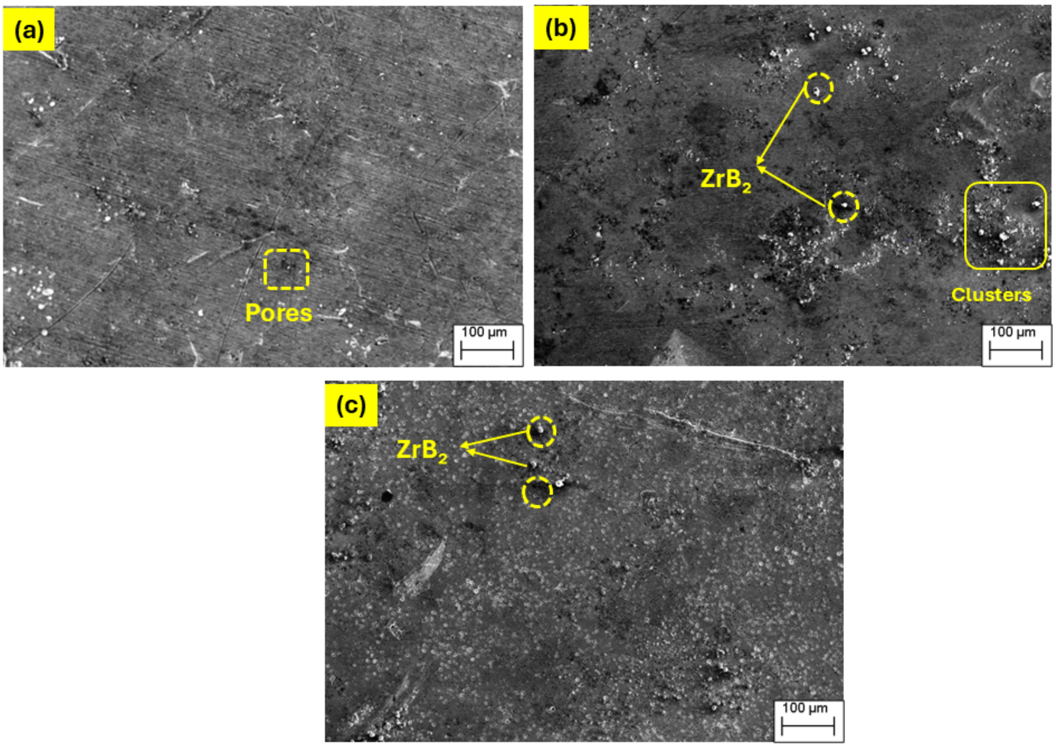

Fig. 1. SEM Images of a) TC0 (Al6061), b) TC1, c) TC2

The incorporation of ZrB_2 nanoparticles into Al6061 alloys tempered in the T4 condition significantly enhances the microhardness of the composite. At a reinforcement level of 1 wt.%, studies have shown an approximate 15% increase in hardness shown in Fig. 2. This improvement is primarily attributed to two key mechanisms: microstructural refinement and effective load transfer. The fine distribution of hard ZrB_2 particles within the aluminum matrix acts as obstacles to dislocation movement, thereby increasing the resistance to indentation and wear. Additionally, the strong interfacial bonding between the ZrB_2 particles and the Al6061 matrix facilitates efficient load transfer during mechanical stress, contributing to the overall hardness of the composite [16].

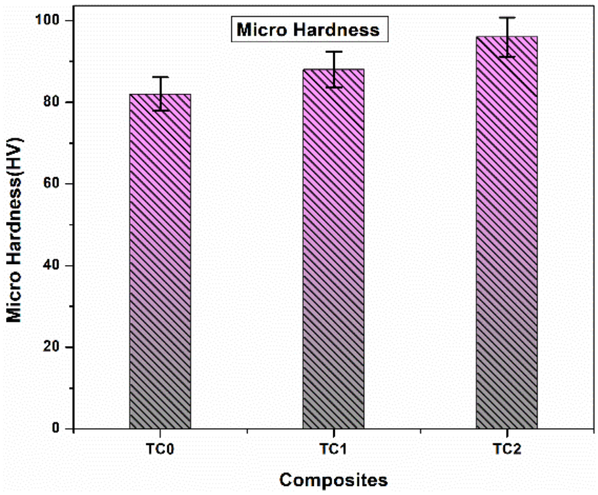

Fig. 2. Micro Hardness of T4 treated nano compositesHowever, this enhancement in hardness is often accompanied by a slight reduction in ductility [16]. As the wt.% of ZrB_2 increases, the aluminum matrix becomes more constrained, reducing its capacity to absorb strain, which slightly compromises ductility. This trade-off between hardness and ductility is a common characteristic in metal matrix composites (MMCs) and highlights the need for optimizing reinforcement content to balance mechanical properties effectively.

3.3. Wear analysis

3.3.1. Effect of load on wear rate

As the normal load applied to Al6061-ZrB_2 composites increases from lower to higher values, such as from 20 N to 40 N, the wear rate correspondingly rises, highlighting the significant impact of load on wear loss . This increase in wear rate is primarily due to the elevated contact stress and intensified frictional interactions at the sliding interface under higher loads. The amplified load causes more substantial deformation of the aluminum matrix and heightens the likelihood of microplowing and micro-cutting actions by abrasive particles or surface asperities. Although the dispersed ZrB_2 particles enhance the composite's wear resistance by bearing part of the load and preventing deep surface damage, excessive normal loads can surpass the protective capacity of these reinforcements shown in Fig. 3 [16]. This results in more pronounced material removal and the formation of deeper wear scars, ultimately contributing to the observed increase in wear rate as the load intensifies. The specific wear rate of the samples has also been calculated from Eq. (1). It can be observed that Specific wear rate decreases with increasing ZrB_2 concentration.

$$\textit{Specific Wear Rate} = \frac{\textit{Volume loss}\left(\mathrm{mm}^3\right)}{\textit{Load applied}\left(\mathrm{N}\right)\times \textit{Sliding distance}\left(\mathrm{m}\right)} \quad (1)$$

3.3.2. Impact of olad on the Coefficient of Friction (CoF)

Interestingly, as the applied load on Al6061-ZrB_2 composites increases, the wear rate tends to rise, while the coefficient of friction (COF) decreases shown in Fig. 4. This counter-intuitive behavior is primarily attributed to two phenomena:

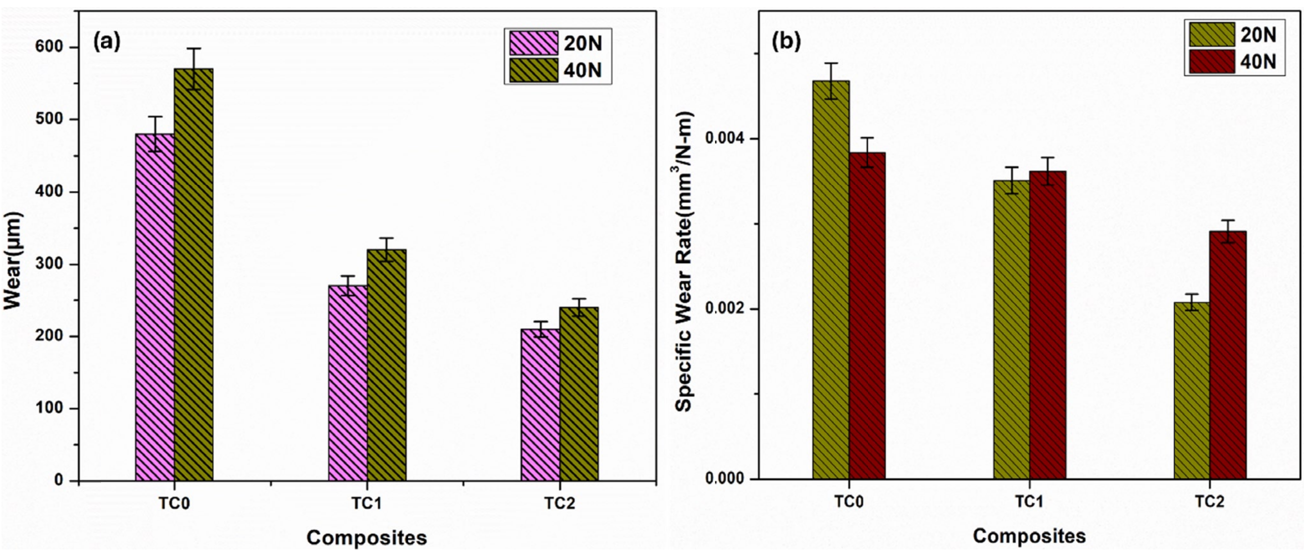

Fig. 3. a) Wear, b) Specific wear rate of the T4 heat treated nano composites at 20 N and 40 N load

thermal softening of the matrix material and the formation of lubricating layers during sliding. When the load is increased, the frictional heat generated at the contact surface intensifies, causing the aluminum matrix to soften. This thermal softening reduces the hardness and rigidity of the surface, making it more susceptible to deformation and smoothing out surface asperities. Consequently, the mechanical interlocking between the composite surface and the counterface diminishes, which lowers the frictional resistance and, subsequently, the COF [17]. Also, the increased load enhances the formation of wear debris, which tends to compact and pile up in the sliding interface. This debris, oxidized and broken-up from the aluminum matrix and ZrB_2 particles, constitutes a thin tribological layer that works as a solid lubricant. This lubricating layer reduces metal-to-metal contact, which also lowers the friction in sliding. Notwithstanding the reduction in COF, the total wear rate continues to rise because the heat-softened aluminum matrix is more susceptible to material loss under the increased load. The combination of thermal softening and lubricating debris layer formation thus accounts for the paradoxical reduction of friction with a rise in wear at higher loads.

At higher loading conditions, Al6061-ZrB_2 composite wear behavior is mainly controlled by adhesive wear and abrasive wear mechanisms. Abrasive wear is observed when hard asperities or wear debris particles move along the composite surface, resulting in cutting or plowing action. In Al6061-ZrB_2 composites, hard ZrB_2 particles introduced significantly improve the abrasion resistance of the material [18]. These particles act as efficient load-supporting sites and function as micro-barriers restricting surface damage through minimizing groove depth and severity due to sliding. Their high hardness also avoids excessive material removal and thus leads to enhanced wear resistance.

Aside from abrasive wear, adhesive wear emerges at high loads. The process is characterized by local welding and transfer of material between the composite and counter surface, often constructed from hardened steel. Micro-welding of asperities under elevated contact pressures and increased friction when sliding is encouraged, causing detachment of particles and material loss from the aluminum matrix. Despite the protective role of ZrB_2 particles, they cannot fully eliminate wear escalation at higher loads. This is due to the localized deformation of the softer aluminum matrix, which can still undergo wear even when reinforced. At very high stresses, particle pull-out or interface cracking may occur if the load exceeds the bonding strength between the ZrB_2 particles and the aluminum matrix, contributing to the formation of wear debris [19,20].

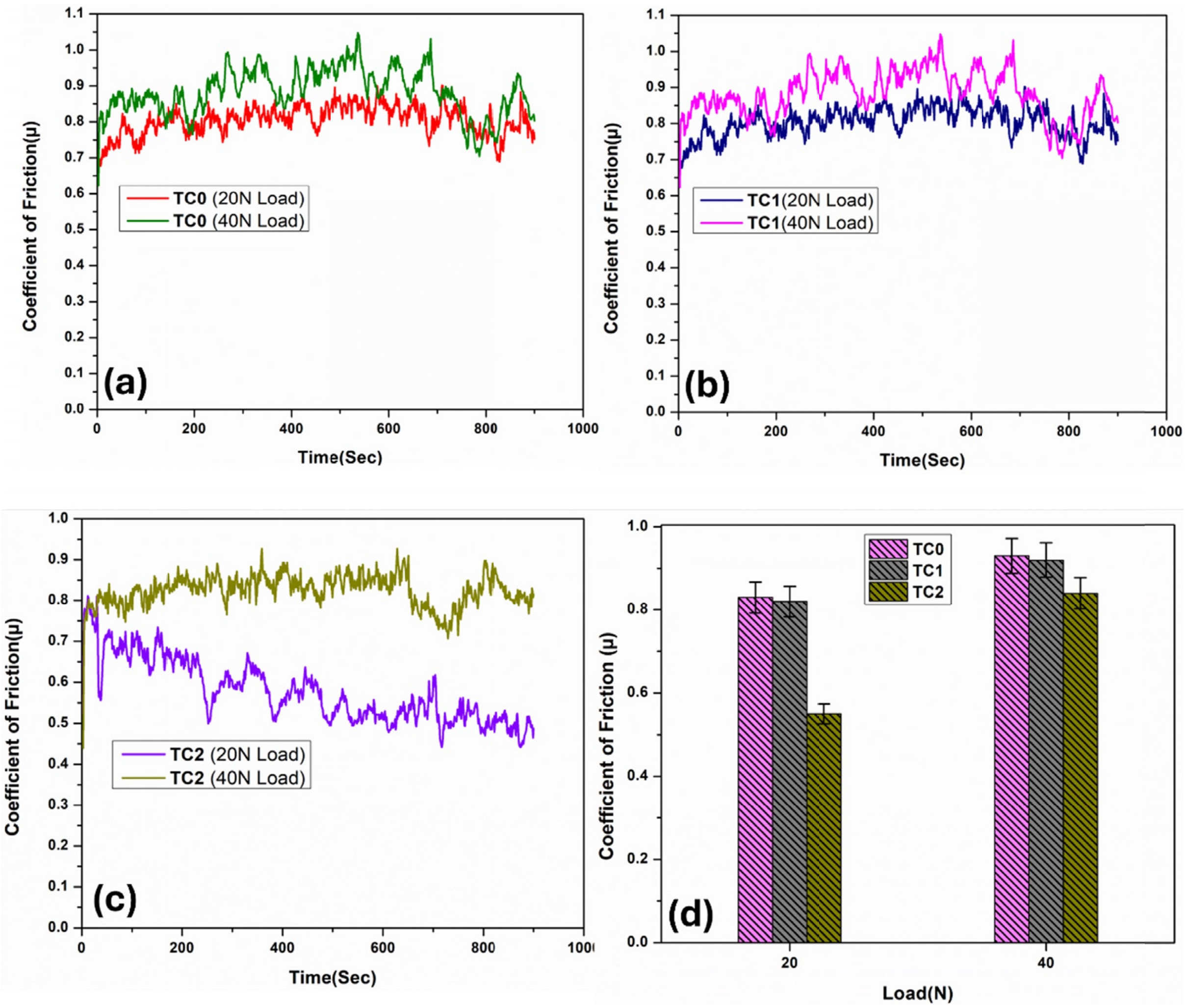

Fig. 4. Coefficient of Friction Vs time curves for Composite a) TC0 (Al6061), b) TC1, c) TC2, d) Coefficient of Friction Vs Load

Overall, while ZrB_2 particles enhance the hardness and wear resistance of the composite, extreme loading conditions still promote wear due to matrix vulnerability and debris generation. This illustrates the critical balance between the reinforcing effect of ZrB_2 and the inherent limitations of the aluminum matrix under high-stress conditions.

3.3.3. Wear morphology

The morphology of wear of Al6061-ZrB_2 composites is remarkably defined by increased surface integrity and domination of gentle abrasive wear processes. Improved wear behavior is mainly contributed through the strengthening effect offered by the homogeneously dispersed ZrB_2 particles in the aluminum matrix. ZrB_2, as a hard ceramic element with superior mechanical properties including high hardness and favorable thermal stability, is responsible for reinforcement of the composite [21]. Upon incorporation into the Al6061 matrix, these particles can support the load during sliding contact efficiently, with a considerable diminution of direct stress on the softer aluminum matrix. This ability to share loads prevents severe surface cuts and unnecessary material loss, thus maintaining the surface integrity of the composite. The major wear mechanism in this composite is mild abrasive wear, in which the hard ZrB_2 particles prevent penetration and cutting by abrasive particles or asperities from the counter surface. As a result, rather than deep grooves or severe surface plowing, the wear scars are shallow and more evenly distributed, reflecting controlled surface abrasion. This mild abrasion reduces material loss and reduces severe surface damage shown in Fig. 5. In addition, the good cohesion between ZrB_2 particles and the aluminum matrix ensures secure embedding of reinforcements during sliding contact. The robust particle-matrix adhesion inhibits particle pull-out and lowers the possibility of micro-cracking, further allowing the composite to improve wear resistance [22].

EDX analysis of the worn surface of the Al6061/ZrB_2 composite indicates that abrasive wear is the predominant mechanism, as evidenced by the displacement of material toward the edges of the wear track, which intensifies surface damage. The presence of oxygen, aluminum, and zirconium on the worn surface further confirms the formation of tribo-oxidation layers, primarily Al_2O_3 and ZrO_2. These oxides serve a dual role: acting as protective barriers that increase surface hardness and simultaneously limiting direct metal-to-metal contact, thereby mitigating wear.

4. Conclusions

- The addition of ZrB_2 nanoparticles in Al6061 increased the tensile strength, microhardness and wear resistance of the composite considerably.

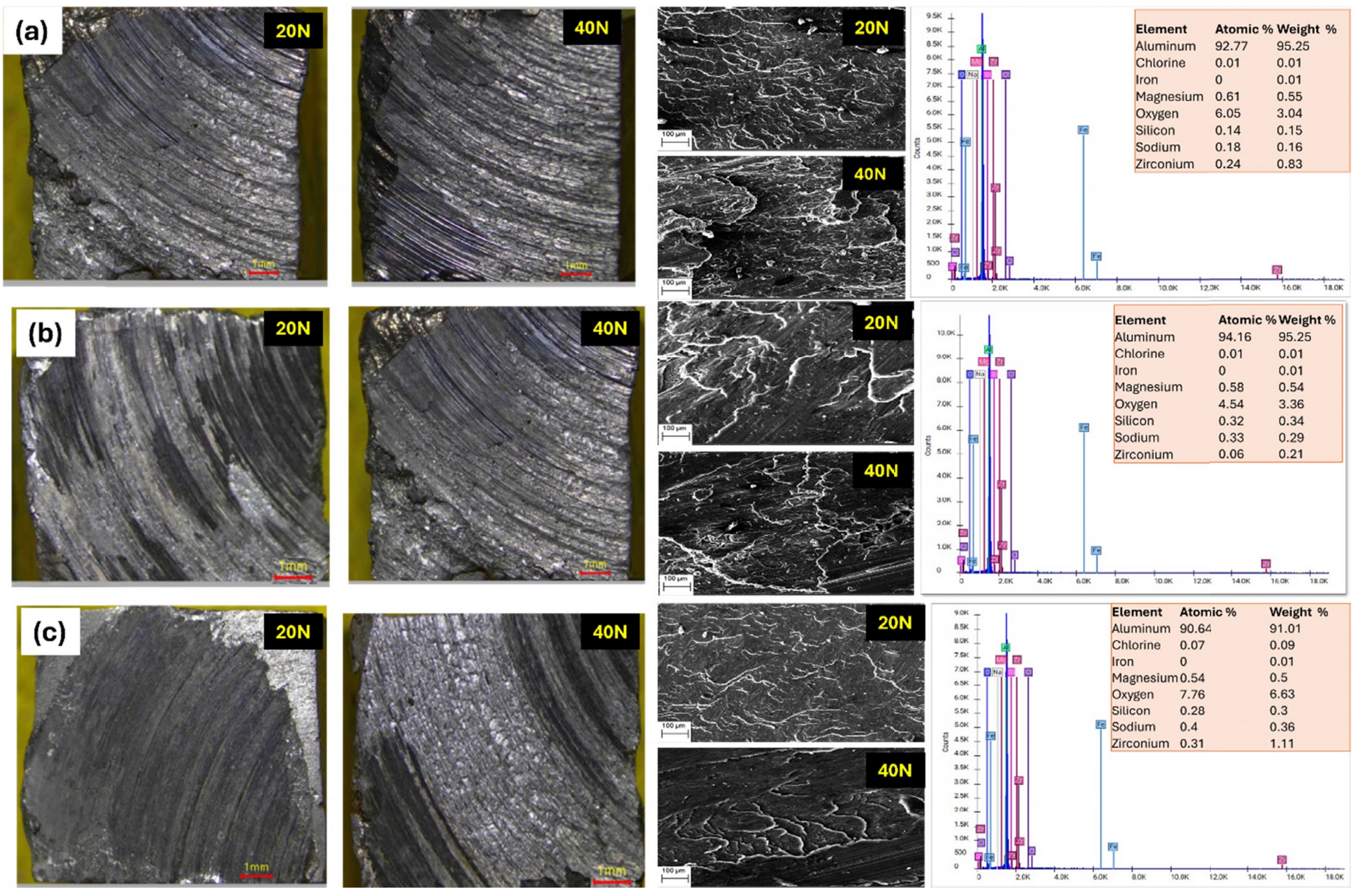

Fig. 5. Wear morphology of a) TC0 (Al6061), b) TC1, c) TC2

- T4 heat treatment, involving solutionizing, quenching, and natural aging, further enhanced the mechanical properties by grain refinement and precipitation hardening.
- Uniform distribution of ZrB_2 particles in the Al6061 matrix ensured good load-sharing under wear, lowering surface stress, material loss and improving the tensile properties of the composites.
- Wear analysis indicated that the composite predominantly exhibited mild abrasive wear mechanisms, with shallow and uniformly distributed wear scars, demonstrating controlled surface abrasion.
- SEM analysis confirmed that the strong bonding between ZrB_2 and the aluminum matrix minimized particle pull-out and micro-cracking, maintaining surface integrity during sliding.
- The developed Al6061-ZrB_2 composites are highly suitable for automotive and aerospace applications, where enhanced surface durability and wear resistance are critical.

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